

FCS20 - Procedure for the Derivatization of GHB

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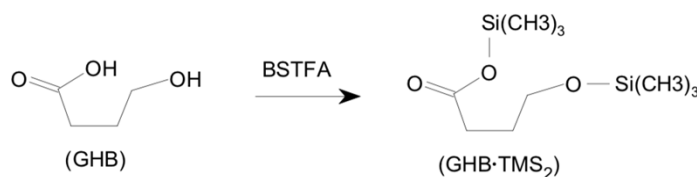
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1. Scope

- 1.1. This procedure is intended to provide instructions on how to derivatize the controlled substance Gamma-Hydroxybutyric Acid (GHB) in standards and test items suspected to contain GHB in the Forensic Chemistry Unit (FCU). This procedure is to be used when GHB is suspected in a test item and either Gas Chromatography Mass Spectrometry (GC-MS) or Gas Chromatography Flame Ionization Detection (GC-FID) will be used for analysis.

2. Background

- 2.1. The analysis of GHB in test samples with GC-MS or GC-FID may result in a false detection of the thermal decomposition product of GHB, Gamma-Butyrolactone (GBL). Legal classification of GHB is different than GBL, requiring the determination of GHB in a select way from GBL.
- 2.2. To prevent the degradation of GHB at the sample inlet of the GC-MS or GC-FID, the chemical GHB is derivatized with N, O-Bistrifluoroacetamide (BSTFA). This derivatization results in a unique signal on the GC-MS and GC-FID that may be easily differentiated from GBL (SWGDRUG, 2005):



- 2.3. The molecular ion GHB-TMS₂ with electron impact ionization (GC-MS) is weak at m/z 248, however, the signal at m/z 233 representing the methyl group cleavage is prominent.
- 2.4. The chemical GBL does not derivatize with BSTFA and typically elutes earlier on a polydimethylsiloxane column.
- 2.5. This method provides documentation for the examination of evidence to conform to the requirements of the Department of Forensic Sciences (DFS) Forensic Science Laboratory (FSL) *Quality Assurance Manual*, the accreditation standards under ISO/IEC 17025 (current revision), and any supplemental standards.

3. Safety

- 3.1. Wear personal protective equipment (e.g., lab coat, gloves, mask, eye protection), when carrying out standard operating procedures.
- 3.2. Read Material Safety Data Sheets to determine the safety hazards for chemicals and reagents used in the standard operating procedures.
- 3.3. Note: BSTFA is highly flammable and shall be stored in a flammable cabinet and prepared in a fume hood.

4. Materials Required

- 4.1. Glass pipettes
- 4.2. Glass vials / sample holders
- 4.3. Ethyl Acetate
- 4.4. Nitrogen evaporation System (TurboVap or equivalent)

5. Standards and Controls

- 5.1. Gamma-Hydroxybutyric Acid, GHB (sodium salt)
- 5.2. N, O-Bistrifluoroacetamide , BSTFA (with or without 1% Trimethylchlorosilane, TMCS)
 - 5.2.1. Note: TMCS may be used to accelerate the derivatization at cooler temperatures.

6. Calibration

- 6.1. Not applicable

7. Procedures

7.1. Drying Procedure

7.1.1. Aqueous samples will be dried down using a nitrogen evaporation system (i.e. TurboVap or equivalent) prior to derivatization

7.1.1.1. Dry the aqueous sample for 30-45 minutes (or until solid is present) with the plate temperature not exceeding 80°C.

7.1.1.2. Keep the gas flow rate between 15-25 psi to minimize splashing.

7.2. Derivatization Procedure

7.2.1. Place the test sample or standard (or extraction thereof) in a glass vial or test tube.

7.2.2. Heat this sample or standard at about 55 °C for about 30min in a fume hood.

7.2.3. Add to this vial or test tube:

7.2.3.1. 100µL ethyl acetate (mix/vortex)

7.2.3.2. 100µL BSTFA (with or without 1% TMCS)

7.2.3.3. Cap immediately and mix/vortex

7.2.4. Allow sample to incubate at room temperature for about 15 minutes.

7.2.5. Transfer contents to GC-MS or GC-FID sample vial for analysis.

8. Sampling

8.1. Perform sampling as per *FCS01 – SOP for Detecting Controlled Dangerous Substances*.

9. Calculations

9.1. Not applicable

10. Uncertainty of Measurement

10.1. Not applicable

11. Limitations

- 11.1. Although it is possible to simultaneously detect GBL with GHB via derivatization of GHB with BSTFA, caution should be used in interpreting GBL due to formation of GBL from excess GHB (i.e., non-derivatized GHB).
- 11.2. BSTFA reacts with any protic solvent; thus, any source of aqueous solvents must be minimized. Use instead non-protic solvents, e.g., chloroform or ethyl acetate when possible.

12. Documentation

- 12.1. FCU Examination Worksheets
- 12.2. FCU Report of Results

13. References

- 13.1. Forensic Science Laboratory Quality Assurance Manual (Current Version)
- 13.2. Gamma-Hydroxybutyrate / Butyric Acid, SWGDRUG, May 16, 2005
- 13.3. Gamma-Hydroxybutyrate (GHB) Quantitation by GC/MS, DC OCME